UNIVERSIDADE FEDERAL DE SERGIPE

PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA

EFEITO DO MOMENTO DE FOTOATIVAÇÃO NAS PROPRIEDADES DE CIMENTOS RESINOSOS EXPERIMENTAIS DE DUPLA ATIVAÇÃO COM DIFERENTES CONCENTRAÇÕES DE CARGA

Aracaju

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ANA CARLA DE ASSUNÇÃO OLIVEIRA

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Dissertação apresentada ao Programa de Pós-graduação em Odontologia, da Universidade Federal de Sergipe, para obtenção do título de Mestre em Odontologia.

Orientador: Prof. Dr. André Luis Faria e Silva

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Ata da sessão de Defesa de Dissertação de Mestrado de **ANA CARLA DE ASSUNCAO OLIVEIRA**

Às quatorze horas do dia vinte e seis de Janeiro de dois mil e quinze, realizou-se no Auditório do CCBBS ,no Campus da Saúde da Universidade Federal de Sergipe, a sessão pública de defesa de dissertação de Mestrado em Odontologia de **ANA CARLA DE ASSUNCAO OLIVEIRA** sob o título: **" EFEITO DO MOMENTO DE FOTOATIVAÇÃO NAS PROPRIEDADES DE CIMENTOS RESINOSOS EXPERIMENTAIS DE DUPLA ATIVAÇÃO COM DIFERENTES CONCENTRAÇÕES DE CARGA"** presidida pelo Prof. Dr. André Luis Faria e Silva, na qualidade de orientador, que por sua vez passou à palavra a candidata para proceder à apresentação do seu trabalho. Logo após, o primeiro examinador, Prof. Dr. Luiz Alves de Oliveira Neto, arguiu a candidata que teve igual período para defesa. O mesmo aconteceu com o segundo examinador, Prof. Dr. Carlos Jose Soares. Em seguida, o Prof. Dr. André Luis Faria e Silva, orientador da candidata, teceu comentários sobre o trabalho apresentado. Encerrada esta etapa, os presentes retiraram-se do recinto, permanecendo apenas a banca examinadora para avaliação. Após esta, a banca decidiu considerar a candidata **APROVADA**. Nada mais havendo a tratar, a presente ata foi lavrada e, depois de lida e aprovada, será assinada pela banca examinadora e pela mestranda.

Aracaju, 26 de Janeiro de 2015

Prof. Dr. André Luis Faria e Silva
Orientador

Prof. Dr. . Luiz Alves de Oliveira Neto
1ª Examinador (FON-USP)

Prof. Dr. Carlos José Soares
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Ana Carla de Assunção Oliveira
Mestranda

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RESUMO

A redução da tensão de contração de cimentos resinosos, sem perdas nas suas propriedades mecânicas, é essencial para a longevidade das cimentações adesivas. Tem sido demonstrado que a fotoativação tardia dos cimentos resinosos duais reduz as tensões de contração. Entretanto, a influência desta postergação da fotoativação sobre as propriedades mecânicas mostrou ser dependente da composição do material. Assim, o objetivo deste estudo foi avaliar o efeito da concentração de carga nas propriedades de cimentos resinosos de dupla ativação fotoativados imediatamente ou após 5 minutos de sua manipulação. Cimentos experimentais duais foram manipulados com matriz monomérica BisGMA/TEGDMA (1:3 em massa) com 60, 65 ou 68% de carga (em massa) de partículas silanizadas de bório-alumínio-silicato. A viscosidade dos cimentos experimentais foi avaliada em viscosímetro digital. Dois protocolos de fotoativação foram adicionalmente avaliados, usando um ponto de fotoativação ou 3 pontos, para isto, usou-se apenas o cimento com 65% de carga. Corpos-de-prova em forma de barra (25 x 2 x 2 mm) foram confeccionados, sendo a fotoativação realizada imediatamente ou 5 minutos após a inserção dos cimentos na matriz (n = 7). Ensaio de flexão de três pontos foi realizado e os valores de resistência flexural e módulo de elasticidade foram mensurados. Os corpos-de-prova fraturados foram embutidos (n = 5) em cilindros de resina acrílica, polidos e a leitura de dureza Vickers foi realizada. Os dados de viscosidade foram submetidos à ANOVA de uma via, enquanto que os dados das propriedades mecânicas avaliadas foram analisados por ANOVA de duas vias. Todas as comparações múltiplas foram realizadas com o teste de Tukey ($\alpha = 0,05$). Amostras fotoativadas em 3 pontos apresentaram maiores valores de resistência flexural. Apenas para a fotoativação em 1 ponto, o momento de fotoativação afetou o módulo de elasticidade (maior para tardia). Os cimentos com 68% de cargas mostraram a maior viscosidade e os com 60% a menor. Independentemente do momento da fotoativação, o cimento com 65% de carga apresentou os maiores valores de resistência flexural e módulo de elasticidade, enquanto que adição de 68% de carga resultou nos menores valores. Já para dureza, o cimento com 68% de carga apresentou os maiores valores, não havendo diferença entre 60 e 65% de carga. Como conclusão, a concentração de carga afeta as propriedades mecânicas dos cimentos e este efeito independente do momento da fotoativação.

Palavras-chave: Cimentos de resina; Materiais dentários; Polimerização.

ABSTRACT

The reduction of shrinkage stress of resin cements, without loss of their mechanical properties, is essential to longevity of adhesive luting procedures. It has been demonstrated that the delayed light-curing of dual-cured resin cements reduces the shrinkage stress. However, the effects of this postponement on mechanical properties showed are dependent form material composition. Thus, the aim of this study was to evaluate the effect of filler content on properties of dual-cured resin cements light-cured immediately or 5 minutes after their mixing. Two light-curing protocols were additionally evaluated, using a single or 3-point of light-activation. For this purpose, only the cement with 65% of filler was used. Experimental dual-cured resin cements were formulated using a monomeric matrix BisGMA/TEGDMA (1:3 in weight) and filled with 60, 65 or 68% of silanated barium borosilicate glass. The viscosity of experimental cements was measured using a digital viscometer. Bar-shaped specimens (25 x 2 x 2 mm) were confectioned, while the light-curing was performed immediately or 5 minutes after the insertion of cements into the mold ($n = 7$). Three-point bending test was performed and the values of flexural strength and elastic modulus were measured. The fractured specimens ($n = 5$) were included into epoxy resin cylinders, polished and the Vickers hardness measurement was performed. Data from viscosity were submitted to one-way ANOVA, while the data from mechanical properties were analyzed by two-way ANOVA. All pair-wise comparison were performed using Tukey's test ($\alpha = 0.05$). Specimens that were light-cured at 3 points showed the highest values of flexural strength. Only for light-activation at 1 point, the moment of light-activation affected the elastic modulus (higher for delayed). Cements with 68% of filler showed highest viscosity and those with 60% presented the lowest one. Irrespective of light-curing moment, the cement with 65% of filler presented the highest values of flexural strength and elastic modulus. Addition of 60% of filler resulted in the lowest elastic modulus, while 68% of filler resulted in lowest flexural strength. Regarding the hardness, the cement with 68% of filler showed the highest values, while there was no difference between 60 and 65% of filler. In conclusion, the filler content affects the mechanical properties of cements and this effect is independent form moment of light-curing.

Key-words: Resin Cements; Dental Materials; Polymerization.

SUMÁRIO

1. INTRODUÇÃO

Os cimentos resinosos são amplamente utilizados na cimentação de restaurações indiretas, principalmente livres de metal, e de pinos intrarradiculares. Neste último caso e em restaurações indiretas com elevada espessura, a luz emitida pelo aparelho fotopolimerizador que alcança o cimento resinoso não é suficiente para desencadear adequada reação de polimerização.[1-4] Assim, cimentos com mecanismo duplo de ativação da reação de polimerização, tanto física (luz) quanto química tem sido desenvolvidos para possibilitar que a ativação química da reação compensasse a ausência (ou redução) da luz emitida pelo fotopolimerizador. Adicionalmente, tem sido demonstrado que a fotoativação destes cimentos duais é importante para aumentar o grau de conversão destes cimentos e, por consequência, de suas propriedades mecânicas.[5-7]

Além dos possíveis problemas relacionados à polimerização dos cimentos, a contração volumétrica dos materiais à base de resina também é um problema que pode afetar os procedimentos de cimentação adesiva.[8,9] Esta contração, que ocorre inevitavelmente, é causada pela substituição de interações intermoleculares por ligações intra-moleculares, que ocupam menor espaço.[8] Esta contração é ainda mais acentuada em cimentos resinosos, quando comparados à resina composta, devido à maior concentração de monômeros diluentes que, por terem menor peso molecular, resultam, comumente, em maior grau de conversão de monômeros em cadeias poliméricas.[10] Considerando que o cimento resinoso está aderido ao substrato dental e ao material restaurador/pino, a deformação do cimento é restrita e tensões sobre as interfaces de união, na estrutural dental e no material restaurador irão ocorrer, podendo prejudicar a longevidade do procedimento restaurador.[8] Esta tensão de contração é ainda maior quando se considera que os cimentos resinosos são utilizados em situações clínicas com alto fator de configuração cavitária (Fator-C)[1].[11]

Dentre os protocolos clínicos que buscam reduzir a tensão gerada pela contração de polimerização de cimentos resinosos duais, a postergação da fotoativação tem demonstrado ser uma técnica efetiva.[12] A mistura das pastas base e catalisadora destes cimentos resulta em início do processo de polimerização pela ativação química, que ocorre de maneira lenta. Quando a fotoativação é feita imediatamente após a mistura, entretanto, a reação é acelerada e o polímero atinge rapidamente elevado módulo de elasticidade. Com o aumento da rigidez (geleificação) do material, o material perde a capacidade de se deformar e dissipar

[1] Razão da área aderida pela área não unida, e que é capaz de dissipar as tensões.

parcialmente as tensões geradas pela contração.[13,14] Já quando a fotoativação é postergada (fotoativação tardia), a fase pré-gel do processo de polimerização é prolongada e, assim, a contração que ocorre após o material apresentar maior rigidez é reduzida.[12]

A fotoativação realizada cinco minutos após a manipulação de cimentos duais resulta em redução da tensão de contração, mas o efeito desta postergação da fotoativação pode afetar negativamente propriedades mecânicas de alguns cimentos.[15] É fundamental que técnicas que possibilitem a redução das tensões de contração não afetem negativamente as propriedades dos cimentos, o que poderia prejudicar a longevidade do procedimento restaurador. Considerando que o efeito da fotoativação tardia em propriedades mecânicas é dependente do material, o conhecimento dos componentes dos cimentos que possam afetar este comportamento é importante. Um destes componentes é a carga inorgânica, sendo que a sua concentração afeta a viscosidade e, por consequência, o comportamento visco-elástico do cimento durante o processo de polimerização.

2. OBJETIVOS

O objetivo deste trabalho foi avaliar os efeitos da concentração de carga em propriedades físico-mecânicas de cimentos resinosos de dupla ativação fotoativados imediatamente ou 5 minutos após sua manipulação.

3. METODOLOGIA

3.1 Delineamento experimental

Este estudo experimental apresenta um delineamento fatorial de 3 x 2 para avaliar os fatores ‘concentração de carga’, em 3 níveis, e ‘momento de fotoativação’ em 2 níveis. Carga inorgânica foi adicionada à matriz resinosa de cimentos resinosos experimentais na concentração de 60, 65 ou 68% em massa. A fotoativação destes cimentos foi realizada imediatamente ou após 5 minutos de sua manipulação. As variáveis respostas foram resistência flexural, módulo de elasticidade e dureza Vickers. A viscosidade dos cimentos também foi mensurada.

3.2 Formulação experimental dos cimentos resinosos duais

A matriz dos cimentos resinosos experimentais foi obtida a partir da mistura dos monômeros 2,2-bis [4-(2-hidroxi-3-metilacriloxipropoxi) fenil]-propano (BisGMA) e dimetacrilato de trietilenoglicol (TEGDMA) na proporção de 1:3 em massa. Todos os monômeros foram obtidos da empresa Esstech Inc. (Essington, PA, USA) e foram utilizados sem purificação adicional. Para a obtenção de pastas base e catalisadora, os seguintes iniciadores, co-iniciadores e inibidores foram adicionados nas seguintes concentrações (em massa):

Pasta Base: 0,8% de canforoquinona (Esstech Inc.) como fotoiniciador, 3,0% de dimetil-p-toluidina (Esstech Inc.) como co-iniciador e 0,2% de hidroxitolueno butilado (Esstech Inc.) como inibidor.

Pasta Catalisadora: 3,0% de peróxido de benzoíla (Vetec, Rio de Janeiro, RJ, Brasil) como iniciador químico e 0,2% de hidroxitolueno butilado (Esstech Inc.) como inibidor.

O fotoiniciador foi adicionado à pasta base em ambiente com luz amarela. Todos os componentes dos cimentos foram pesados em balança analítica digital com precisão de 0,01mg (AUW220D, Shimadzu Corporation, Tóquio, Japão). Após a pesagem dos componentes, a homogeneização foi realizada por meio de vibração mecânica de baixa frequência por um período de 30 minutos. Nas pastas base e catalisadora foram incorporadas partículas silanizadas de vidro de bário-boro-silicato com diâmetro médio de 2.0 µm com 60%, 65% ou 68% em peso (Figura 1A e 1B).

A definição das concentrações de carga foi determinada a partir de estudo piloto, assim como a proporção monomérica de 1:3. Neste estudo, considerou-se a viscosidade e a característica de manipulação dos cimentos experimentais.

A incorporação das partículas inorgânicas foi realizada por meio de misturador mecânico de alta frequência (Figura 2A e 2B) por duas vezes, a 2300 rpm durante 20 segundos cada. Os cimentos experimentais formulados foram acondicionados em frascos vedados (Figura 2C) e em seguida, armazenados em ambiente livre de luz sob refrigeração a 4° C.

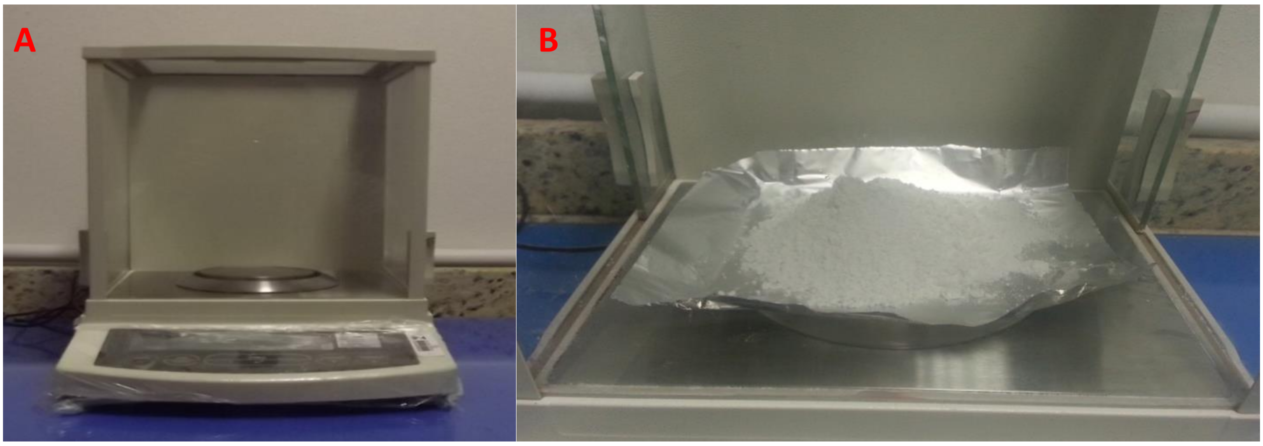

Figura 1. A: Balança digital utilizada para pesagem dos componentes dos cimentos.
B: Pesagem das partículas silanizadas de vidro de bário-boro-silicato.

Figura 2. A e B: Misturador mecânico de alta frequência (SpeedMixer). C: Frasco vedado para armazenamento dos cimentos.

Os cimentos experimentais foram manipulados no Centro de Desenvolvimento e Controle de Biomateriais, vinculado à Faculdade de Odontologia da Universidade Federal de Pelotas, Pelotas, RS.

3.3 Viscosidade

A mensuração da viscosidade dos cimentos experimentais foi realizada por meio de viscosímetro digital LVDV-II+Pro (Brookfield, Milddleboro, MA, EUA) - Figura 3A e 3B. A análise da viscosidade foi feita no modo oscilatório com os seguintes parâmetros: temperatura de 20°C, velocidade 5 rpm, taxa de cisalhamento de 10 Hz com uma varredura de 60 segundos. Três leituras foram realizadas para cada cimento resinoso. Esta mensuração foi realizada no Laboratório de Caracterização de Biomateriais, vinculado ao Departamento de Odontologia, da Universidade Federal de Sergipe.

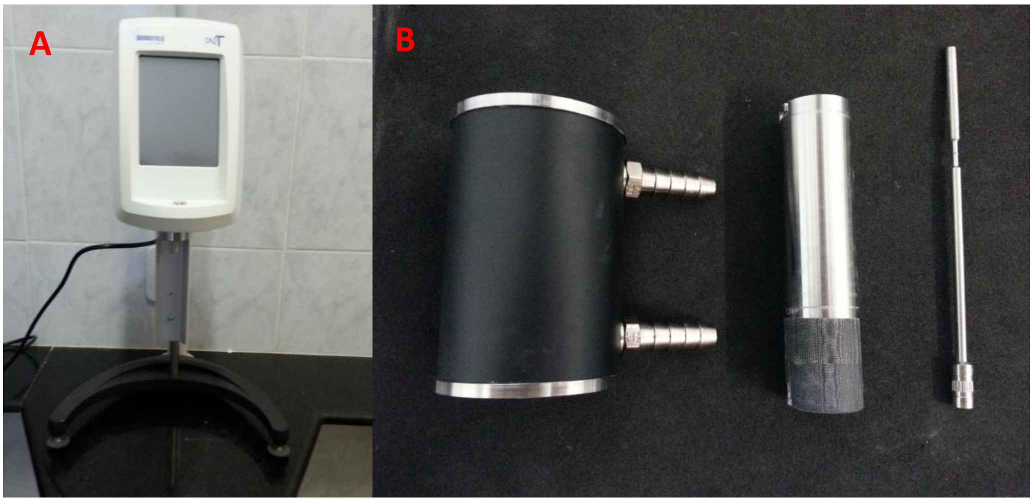

Figura 3. A: Viscosímetro. B:. Componentes do viscosímetro. Na sequência, da esquerda para direita: adaptador da câmara, câmara para armazenamento da amostra e spindle.

3.4 Definição do protocolo de fotoativação

Para as dimensões dos corpos-de-prova para o teste de resistência flexural, seguindo a especificação ISO 4049 (*International Standard Organization Speification* 4049: *Dentistry – polymer-based filling*), está que a fotoativação seja feita em 3 pontos equidistantes sobre o corpo-de-prova. Entretanto, considerando que o momento da fotoativação é um fator em estudo, foi necessário a adaptação do protocolo de fotoativação. Para avaliação de protocolos alternativos, utilizou-se apenas um cimento resinoso (com 65%) e dois modos de fotoativação (tardio e imediato). Para isto, o cimento foi inserido em matriz retangular (25 mm de comprimento x 2 mm de largura x 2 mm de espessura) – Figura 4; e tira transparente de poliéster foi posicionada sobre as superfícies tanto no topo quanto base da amostra para aumentar a lisura da amostra.

Figura 4. Matriz metálica utilizada para confecção das amostras do ensaio de flexão.

Dois protocolos de fotoativação foram avaliados, utilizando um ponto ou 3 pontos. No protocolo de um ponto, a ponta do aparelho fotopolimerizador a base de LED (Radii-Cal; SDI, Bayswater, Victoria, Austrália) foi posicionado a 10 mm da amostra, de forma que o feixe de luz ficasse centralizado sobre a mesma (Figura 5A). Neste modo, a fotoativação das amostras foi realizada por 180 segundos. Buscando maior homogeneidade de polimerização, três aparelhos (Radii-Cal) foram posicionados em pontos equidistantes sobre a amostra e ativados simultaneamente, com as pontas distando 10 mm da amostra (Figura 5B). Neste modo, a fotoativação foi realizada por 60 segundos imediatamente. Para os dois protocolos (um ou 3 pontos), a fotoativação foi realizada imediatamente ou após 5 minutos da inserção do cimento na matriz. A fotoativação da superfície topo, a amostra era removida e nova fotoativação realizada na base pelo mesmo tempo (n = 7).

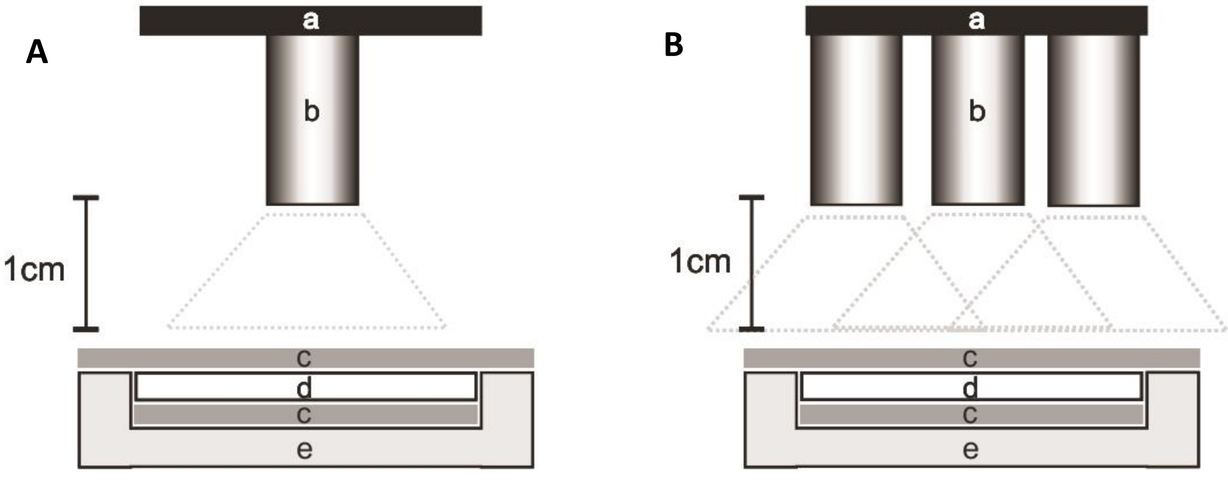

Figura 5. Ilustração esquemática dos protocolos de fotoativação. A) Fotoativação em um ponto; B) – Fotoativação em 3 pontos. a: suporte dos fotopolimerizadores; b: fotopolimerizadores; c: tira de poliéster; d: cimento resinoso; e: matriz metálica.

3.5 Resistência flexural e Módulo de elasticidade

Após a fotoativação, os corpos-de-prova foram removidos e o acabamento realizado manualmente com lixa de Carbeto de Silicio (SiC) de granulação 1200, sob refrigeração a água. As dimensões de cada corpo-de-prova foram conferidas com paquímetro digital (Mitutoyo Corporation, Tóquio, Japão) com precisão de 0,01 mm. O teste de flexão de três pontos foi realizado em máquina de ensaios mecânicos (Instron 3367, Instron Corp., Canton, MA, USA) com velocidade de 0,5 mm/minutos apoiada num dispositivo contendo dois suportes metálicos distantes 20 mm entre si com célula de carga de 1000N (Figura 6). A resistência à flexão foi calculada, σ, em megapascal (MPa) pela equação: $\sigma = 3Fl / 2bh^2$, onde F = carga máxima para fratura da amostra, em newtons, l = distância em milímetros entre os suportes, b = largura, em milímetros no centro da amostra e h = altura, em milímetros, no centro da amostra.

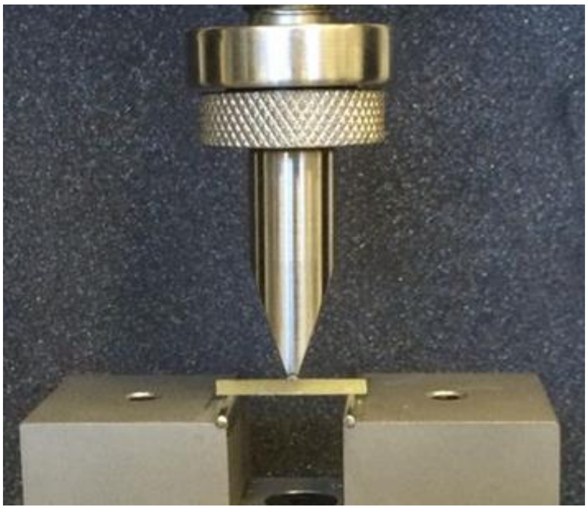

Figura 6. Realização do teste de resistência flexural de três pontos com a barra de cimento resinoso em posição.

O teste de resistência a flexão foi monitorado pelo *software* (Bluehil 2, Instron Co.,Canton, MA, USA) com um computador conectado à maquina de ensaios, o qual gera automaticamente um gráfico de tensão x deformação de cada corpo-de-prova. O módulo de elasticidade (E) foi calculado em gigapascal (GPa) a partir da inclinação da porção linear da curva tensão x deformação, correspondente a deformação elástica do material, utilizando a fórmula: $E = F_1 l^3 / 4bdh^3$, onde F_1 representa a carga em Newtons exercida na amostra, l, distância em milímetros entre os suportes, h a altura da amostra em milímetros, b, a largura da amostra em milímetros e d a deflexão correspondente à carga F_1.

Os dados do estudo para definição do protocolo de fotoativação foram submetidos a ANOVA de dois fatores (protocolo x momento de fotoativação) e teste de Tukey (α = 0,05). Baseado nos resultados deste estudo (página 11), definiu-se que o protocolo com 3 pontos de fotoativação simultâneos era o mais adequado para o experimento de avaliação do efeito da carga nas propriedades mecânicas dos cimentos de duplas ativação fotoativados imediata ou tardiamente. Assim, setes corpos-de-prova foram confeccionados para cada condição experimental (item 3.1) e submetidos ao ensaio de resistência flexural, sendo também mensurado o módulo de elasticidade, da mesma forma descrita anteriormente.

3. Microdureza Vickers

Amostras fraturadas do ensaio de flexão foram embutidas em cilindros de poliestireno com resina acrílica (Figura 7). Cinco corpos-de-prova foram utilizados para cada condição experimental. Os corpos-de-prova foram submetidos à acabamento por meio de lixa de Carbeto de Silício (SiC). As lixas foram utilizadas em granulação decrescente (180, 320, 400, 600 e 1200), sob-refrigeração à água, até a obtenção de um polimento satisfatório para a leitura da microdureza. A dureza Vickers (VHN) foi mensurada em microdurômetro HMV-2 (Shimadzu, Tokyo, Japan) com procedimento automático de aplicação de 9,8N de carga durante 15 segundos, utilizando um endentador Vickers. As endentações foram realizadas no centro das amostras evitando as extremidades. As diagonais das endentações foram mensuradas por meio do cursor do durômetro e, automaticamente, convertidas em valores de dureza Vickers. Seis endentações foram realizadas por corpo-de-prova, sendo a média destas endentações usada na análise estatística dos dados.

Figura 7. Amostras fraturadas embutidas em cilindros de poliestireno com resina.

Todos os ensaios mecânicos foram realizados no Laboratório de Ensaios Mecânicos, vinculado ao Programa de Pós-Graduação em Ciências e Engenharia de Materiais, da Universidade Federal de Sergipe.

4. Análise dos dados

Os dados de cada mensuração (exceto viscosidade) foram, individualmente, submetidos a ANOVA dois fatores e teste de Tukey ($\alpha = 0,05$). Os fatores avaliados foram: ‘concentração de carga’ (3 níveis) e ‘momento da fotoativação’ (2 níveis). Os dados de viscosidade foram submetidos à ANOVA um fator (concentração de carga) e teste de Tukey ($\alpha = 0,05$).

4. RESULTADOS

ARTIGO 1

An alternative specimen preparation technique for 3-point bending tests on dual-cured dental resin cements

Short title: Alternative specimens for 3-point bending test

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Summary

The proper mechanical properties of resin cements are essential to the longevity of indirect restoration, whereas the 3-point bending test is recommended for measuring the flexural strength. The ISO 4049 specification requires light-curing of specimens in three consecutive points; however, this approach cannot be used for dual-cured resin cements. The aim of this study was to investigate the effect of two different specimen preparation techniques on the flexural strength and elastic modulus of experimental dual-cured resin cements immediately or 5 after minutes light curing. Experimental dual-cured resin cements were formulated, and the specimens of these cements were confectioned with the dimension of ISO 4049 specification. Light-activation was performed at one or three points immediately or 5 minutes after the insertion of cement into the matrix (n=7). The three-point bending test was performed and the values of the flexural strength and elastic modulus were recorded. Data were individually analyzed using 2-way ANOVA followed by the Tukey's post hoc test ($P<0.05$). Regardless of the points of light-activation, the specimens that were light-cured at 3 points showed the highest values of flexural strength. Only for light-activation at 1 point did the moment of light-activation affect the elastic modulus, whereas delayed light-activation had the highest values. In conclusion, the number of light-curing points on specimen preparation for the 3-point bending test seems to affect the mechanical properties of dual-cured resin cements.

Keywords: compressive strength, polymerization, resin cements.

Introduction

Dual-cured resin cements are commonly used to lute indirect restorations and intra-radicular dental posts with the aim of combining the advantages of chemically and light-cured polymer-based materials (1). The rationale is to have a material that combines extended working time with capacity for reaching proper polymerization in either the presence or absence of light. Proper polymerization is related to improved mechanical properties and may ultimately impact the longevity of the restorative procedures (2). One of the more commonly used laboratory tests for evaluating the mechanical properties of dental resin cements is the 3-point bending test, which is a useful test for determining the flexural strength and flexural modulus (elastic modulus) of the materials (3-5). These mechanical properties are largely used for characterizing dental materials.

The International Organization for Standardization (ISO) specification 4049 establishes the standards for performing the 3-point bending test for polymer-based dental materials (6). The test consists of the application of a compressive load until there is failure on the center of a bar-shaped specimen that is 25 mm in length, 2 mm in height and 2 mm in width. The bar specimen is supported by two rods, 2 mm in diameter and mounted parallel with a 20 mm span between the supports; meanwhile, the load is applied through a rod that is 2 mm in diameter. This standardization establishes that the specimen fabrication should be performed with the light-cured resin materials inserted into a mold and then followed by light-activation in three consecutive, overlapping areas in both sides of the specimen. This approach is necessary because the length of the specimens is larger than the diameter of the light-curing unit guide. The center of the specimen receives the first light exposure, which is followed by two complementary exposures at equidistant points from the center.

Although the ISO 4049 specification is largely used for testing composites that are light-cured only, the technique recommended for specimen preparation was not appropriate for testing dual-cured resin-based materials. By the time dual-cured materials are inserted into the mold, the polymerization reaction has already started via chemical activation. Therefore, each light-activation is performed over areas of the polymer, presenting with differences in the degrees of C=C conversion. In addition to the possibility of generating a heterogeneous polymer, this approach impairs studies that evaluate the effect of the moment of light-activation (immediately to a few minutes after, for instance) on the mechanical properties of the dual-cured materials (7). Using single light-activation in the center of the specimen with the tip of the light-curing unit, away from the composite, is an alternative. However, to provide light exposure covering the entire bar specimen, a reduction in light irradiance in areas away from the center is expected. Another alternative is to perform light-activation at three equidistant points simultaneously using three light-curing units. To the authors' knowledge, this simple approach has not yet been reported.

The aim of this study was to investigate the effect of light-curing methods using exposure either at a single area, at the center of the bar, or three simultaneous light exposures, covering the entire bar on the flexural strength and elastic modulus of dual-cured resin cements. We tested the hypothesis that the use of the method of three simultaneous light exposures affects the flexural strength and flexural modulus data.

Materials and Methods

Study design

This investigation was conducted using a 2 × 2 factorial study design to evaluate the 'light-curing method' in two levels (single exposure at the center or three simultaneous exposures along the bar) and 'moment of light-curing' of an experimental dental resin cement in two levels (immediately or 5 min after inserting the cement into the mold). The response variables evaluated were the flexural strength (σ_f) and flexural modulus (E_f) obtained through a 3-point blending test.

Formulation of the experimental dual-cured resin cement

A model dual-cured resin luting agent was formulated using the monomers 2,2 – bis[4–(2-hydroxy-3-methacryloxyprop-1-oxy)pheny1]propane (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) at a 3:1 mass ratio. The monomers were obtained from Esstech Inc. (Essington, PA, USA). Silanated barium borosilicate glass fillers, 2 µm in average diameter (Esstech Inc.), were added at 65 mass%. The cement was consisted of two pastes, one labelled base paste and another labelled catalyst paste. Camphorquinone (0.8 mass%) and diethanol-*p*-toluidine (3 mass%), both from Esstech Inc., were added to the base paste as the photoinitiator and co-initiator. Benzoyl peroxide (Vetec, Rio de Janeiro, RJ, Brazil) was added to the catalyst paste at a 3 mass% as self-activated initiation system. Butylated hydroxytoluene (0.2 mass%) was added to both pastes as a radical scavenger.

Specimen preparation for the 3-point bending test

Specimens were prepared according to the bar-shaped dimensions specified by the ISO 4049 standard (6). Equal volumes of base and catalyst pastes were mixed for 15 s and inserted into a metallic split mold with 25 mm in length, 2 mm in width and 2 mm in height. The material was covered by an acetate strip and light-activation was performed using identical light-emitting-diode (LED) light-curing units (Radii-Cal; SDI, Bayswater, Victoria, Australia) with 1200 mW/cm^2 irradiance each.

For the light-activation using a single light exposure area, the tip of the light-curing unit was fixed 1 cm away from the mold and positioned at the center of the specimen for the polymerizing light to reach the entire bar. Light-activation was performed for 180 s. For light-activation using three simultaneous light exposures, three light-curing units that were used at the same time were also positioned 1 cm away from the specimen, but they were distributed at equidistant points from the center of the bar (Figure 1). Light-activation was performed for 60 s to generate the same radiant exposure of the method using a single light exposure area. For both light-curing methods, light-activation was performed immediately or 5 min after inserting the resin cement into the mold. The light-activation procedures were performed at both the top and bottom sides of each specimen; therefore, the total radiant exposure for each specimen was 43.2 J/cm^2. The cured specimens were wet-polished with #1200-grit SiC papers and stored in distilled water at 37±1°C for 24 h in the dark.

Three-point bending test

The dimensions of the bars were checked with a digital caliper accurate to 0.01 mm (Mitutoyo Corporation, Tokyo, Japan). The specimens were positioned in a 3-point bending device coupled to a mechanical testing system (Instron 3367, Instron Corp., Canton, MA, USA). The distance between supports was 20 mm and the load was applied to the center of specimen. The diameter of both supports and of the loading rod was 2 mm.[9] The tests were performed at a crosshead speed of 0.5 mm/min until failure and was monitored by the testing machine software (Bluehill 2, Instron Corp.). To calculate σ_f (MPa), the following equation was used:

$$\sigma_f = \frac{3Fl}{2bh^2} \quad \text{(Eq. 1)}$$

where F is the maximum load (N) exerted on the specimen, l is the distance (mm) between the supports, and b is the width (mm) and h the height (mm) at the center of the specimen. The E_f was calculated using the following equation:

$$E = \frac{F_1 l^3}{4bdh^3} \quad \text{(Eq. 2)}$$

where F_1 is the load (N) exerted on the specimen and d is the deflection corresponding to the load F_1. Data for the σ_f and E_f were individually submitted to 2-way analysis of variance. All pairwise, multiple comparison procedures were performed using the Tukey's method (α = 0.05).

Results

The results for σ_f are shown in Table 1. The statistical analysis revealed a significant effect only for the factor 'light-curing method' ($P < 0.001$), while the factor 'moment of light-curing' ($P = 0.646$) and interaction between the factors ($P = 0.483$) were not significant. The results were expressed as pooled averages for both moments of light-curing. The σ_f was significantly higher for the light-curing method using three simultaneous exposures.

Results for the E_f are shown in Table 2. The statistical analysis showed a significant effect for the factor 'light-curing method' ($P = 0.038$) and for the interaction between factors ($P = 0.013$). The factor 'moment of light-curing' ($P = 0.94$) was not significant. Differences between light-curing methods were observed only for the delayed light-activation procedure, whereas the method that used three simultaneous exposures had the highest values. Irrespective of the light-curing method, no significant difference was observed between the moments of light-activation.

Discussion

The findings of this study demonstrated that the number of light-curing points significantly affected the σ_f of the dual-cured resin cement, irrespective of the moment of light-activation. Light-curing the samples in 3 points resulted in almost 2-fold higher σ_f compared to light-curing in a single point. For the measured values of E_f, the number of light-curing points only affected the values 5 minutes after light-curing was performed, whereas 3-point light-curing increased the elastic modulus. As a result, the hypothesis of study was accepted.

Despite the presence of a polymerization reaction activated by a chemical reaction, several studies have demonstrated that dual-cured resin cements require light-curing to improve their polymerization potential and mechanical properties (1,7-10). In this study, we used a light-curing device tip with an approximately 8-mm diameter, whereas the ISO 4049 specification recommends samples with a 25-mm length. Considering that the light beams emitted by device tip are divergent, the diameter of light over the samples tends to increase with longer distances from the tip (11). However, increasing the distance between the tip and sample also reduces the energy density (12). Attempting to polymerize the sample with single-point light-curing, the tip of the light-curing device was positioned 10 mm from the sample in the present study. A reduction of approximately 75% in the energy density when the tip is positioned at 10 mm from the sample has been reported (12). This significant reduction in the energy density reduces the polymerization of resin cement and its mechanical properties, explaining the lowest values observed when only one point was used to light-cure the samples (13-15). The use of the other two additional points for light-curing increases the energy available to polymerize the resin cement. Additionally, 3-point light-curing reduces the inhomogeneous irradiance profile of irradiance provided by the light-curing device, resulting in more proper polymerization of cement (12,16,17). However, it is important to emphasize that the values of the σf (approximately 65 MPa) reached by samples that were light-cured in a single point was superior to the minimum values required by ISO standardization (50 MPa) (6).

Interestingly, the number of light-curing points only affected the E_f of resin cement for the delayed light-activation. Delayed light-activation has been advocated to slow the polymerization reaction and reduce the polymerization stress of dual-cured resin cements (18,19). The slower chemical polymerization in the first minutes allows for an increase in the duration of the pre-gel polymer stage, resulting in increased flow of the cement and reduced polymerization stress (20-22). Therefore, delaying the light-activation of dual-cured cements allows for relief of this stress, whereas the light-activation is performed when there is a significant conversion of materials (20). It has been demonstrated that the slowest polymerization reaction may results in polymers with reduced elastic modulus (21,23,24). However, no significant differences were observed in the moments of light-activation, irrespective the number of light-curing points.

An important observation of the outcomes in the present study was that the number of light-curing points affected the E_f for only the delayed light-activation mode. A reasonable explanation for these findings can be related to the molecular mobility of reactional media in the moment of light-activation. For immediate light-curing, only a small number of resin monomers react in the moment of light incidence and the high mobility of reactional media allows for achieving additional polymerization even for a low energy density (25). By contrast, higher conversion is expected 5 minutes after mixing resin cement with reduced mobility reaction media. Therefore, a higher energy density can be required to promote a significant improvement in the reactive sites. A higher number of polymerization reactive sites has been related to increased elastic modulus (21,23,24).

In the present study, the sample preparation method for the 3-point bending test significantly affected the mechanical properties of dual-cured resin cements. The alternative method suggested in this study is simultaneous light-curing of the sample in three different points, resulting in increased σ_f. This method also increases the E_f of cements that are light-cured after 5 minutes of mixing. According to ISO 4049 specification, this method uses 3 points of light-curing, allowing for a more homogeneous polymerization. Furthermore, light-curing in distinct points at the same time permits the evaluation of dual cured resin-based materials.

Summary in Portuguese

Cimentos resinosos com propriedades mecânicas adequadas são essenciais para a longevidade de restaurações indiretas, sendo que o ensaio de flexão de 3 pontos é recomendado para mensurar a resistência flexural. A especificação ISO 4049 requer a fotoativação das amostras em três pontos consecutivos, entretanto, esta abordagem não pode ser usada para cimentos resinosos duais. O objetivo deste estudo foi investigar o efeito e duas diferentes técnicas de preparo de amostras na resistência flexural e módulo de elasticidade de cimentos resinosos duais experimentais fotoativados imediatamente ou após 5 minutos. Cimentos resinosos duais experimentais foram formulados, e amostras destes cimentos foram confeccionadas com as dimensões da especificação ISO 4049. A fotoativação foi realizada em um ou três pontos imediatamente ou após 5 minutos da inserção do cimento na matriz (n=7). O teste de flexão de três pontos foi realizado e os valores de resistência flexural e módulo de elasticidade mensurados. Os dados foram individualmente analisados por ANOVA de dois fatores seguido pelo teste de Tukey ($P<0,05$). Em relação aos pontos de fotoativação, as amostras que foram fotoativadas em três pontos mostraram os maiores valores de resistência flexural. Apenas para a fotoativação em um ponto, o momento de fotoativação afetou o módulo de elasticidade, sendo que a fotoativação tardia apresentou maiores valores. Em conclusão, o número de pontos de fotoativação no preparo das amostras para teste de flexão de 3 pontos parece afetar as propriedades mecânicas dos cimentos resinosos.

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Tables

Table 1. Means (SD) of flexural strength in MPa (n=7).

Light-curing method	Moment of ligh-activation		Pooled averages
	Immediate	Delayed	
Single exposure	65.0 (11.3)	63.3 (12.6)	64.2 (11.2) B
Three simultaneous exposures	108.4 (25.8)	116.3 (15.6)	112.3 (20.9) A

For pooled averages, distinct letters indicate significant differences ($P \leq 0.05$).

Table 2. Means (SD) of the flexural modulus in GPa (n=7)

Light-curing method	Moment of ligh-activation	
	Immediate	Delayed
Single exposure	7.6 (0.3) A.a	7.0 (0.4) A.b
Three simultaneous exposures	7.6 (0.8) A.a	8.4 (1.2) A.a

Distinct letters (uppercase for line, lowercase for column) indicate significant differences ($P \leq 0.05$).

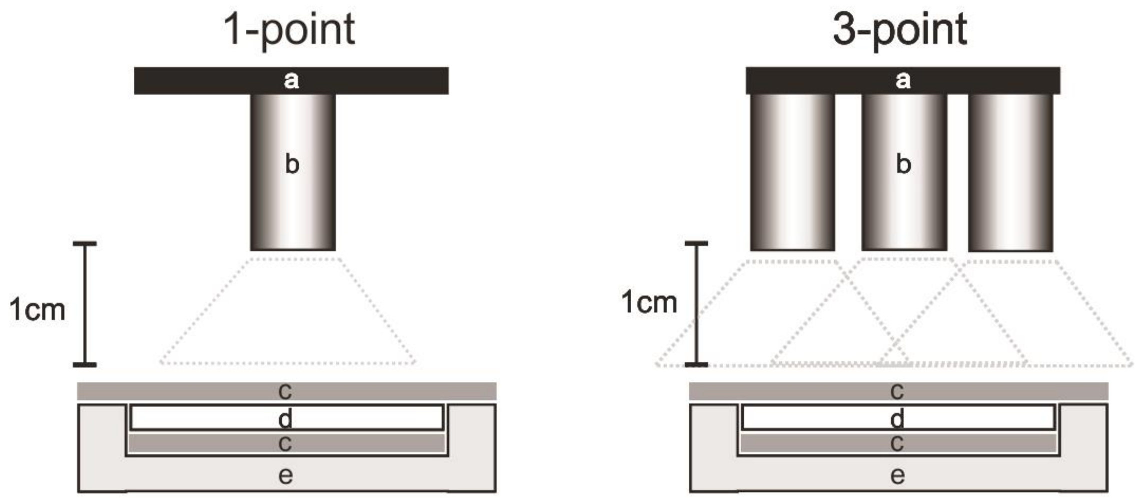

Figure 1. Schematic illustration of the light-activation procedures during specimen preparation. a – Hold to standardize the tip position of light-curing unit; b – Tip of light-curing unit; c – Acetate strips; d – Resin cement; and e – Mold.

ARTIGO 2

Delayed light-curing and mechanical properties of dual-cured cements: effects of filler content

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Abstract

The aim of this study was to investigate the effect of filler content on mechanical properties of experimental dual-cured resin cements immediately or 5 minutes after light curing. Experimental dual-cured resin cements were formulated with 60, 65 or 68% of filler. The viscosity of experimental cements was measured using a digital viscometer. Bar-shaped specimens (25 x 2 x 2 mm) were confectioned, while the light-curing was performed immediately or 5 minutes after the insertion of cements into the mold (n = 7). A three-point bending test was performed and the values of flexural strength and elastic modulus were measured. The fractured specimens were included in epoxy resin cylinders and polished, and the Vickers hardness measurement was performed. Data from viscosity were submitted to one-way ANOVA, while the data from mechanical properties were analyzed by two-way ANOVA. All pair-wise comparisons were performed using Tukey's test ($\alpha = 0.05$). Cements with 68% of filler showed the highest viscosity and those with 60% the lowest. Irrespective of light-curing moment, the cement with 65% of filler presented the highest values of flexural strength and elastic modulus. The addition of 60% of filler resulted in the lowest elastic modulus, while 68% of filler resulted in lowest flexural strength. Regarding the hardness, the cement with 68% of filler showed the highest values, while there was no difference between 60 and 65% of filler. In conclusion, the filler content affects the mechanical properties of cements and this effect is independent from moment of light-curing.

Keywords: Dental cements; Photoactivation; Polymerization; Resin luting agents.

Introduction

Resin cements are largely used on luting procedures of metal-free indirect restorations and fiber posts (1-3). Additionally to obtainment of proper bond strength to dental substrate, improved mechanical properties of these luting agents are essential to longevity of restorative procedures (4). During the cementation of posts and thick indirect restorations, exposed marginal areas can benefit greatly from photo activation, as they are readily accessible to the curing light; however, a significant reduction in its intensity might may significantly decrease occur due to reflecting and scattering effects (5-8). Considering this difficulty to light-cure the resin cements in several clinical conditions, dual-cured resin cement has gained popularity for adhesive luting procedures. The rationale of dual-cured cements is a material capable of reaching a high degree of conversion in either the presence or absence of light, while presents extended working time. Despite this rationale, several studies have demonstrated that light-curing is essential to improve the mechanical properties of dual-cured resin cements (9-11).

In addition to the possible difficulty in light-curing resin cements, the polymerization shrinkage of these materials is another clinical problem that can compromise the longevity of restorative procedures. Unavoidably, the polymerization of all resin-based materials results in volumetric reduction due the replacement of intermolecular interaction by shorter intra-molecular bonds (12). Bonding the resin-based materials to dental substrate and restorative material/fiber posts restricts the shrinkage of material and results in stress on bonding interfaces (12). This shrinkage stress can disrupt the interfaces or generates cracks in the dental tissue or restorative material. The effects of shrinkage stress are more pronounced for resin cement once these materials are used in situations with a high C-factor[2] (13).

One clinical approach used to reduce the deleterious effects of polymerization shrinkage is the delay of light-curing of dual-cured resin cements (14). The elastic modulus and viscosity of cements increases gradually during the polymerization reaction (15). Considering that the chemically-activated reaction is slower than the light-cured reaction, the postponement of the light-curing procedure delays the acquisition of a high elastic modulus (15,16). This allows the polymer chains to rearrange and accommodate the shrinkage by plastic deformation (15). However, in addition to reduced shrinkage stress reduction, the maintenance of the mechanical properties of resin cement is also important in the clinical performance of indirect restorations. A study demonstrated that delaying the light-activation

[2] The C-factor refers to ratio of bonded to non-bonded area in a cavity preparation.

for five minutes can negatively affect the mechanical properties of dual-cured resin cements, while this effect is material-dependent (17).

Several components of resin cements are able to affect their behavior during the polymerization, including the filler content (12,19). The filler content affects the viscosity of resin cements and, consequently, the mobility of reactional media. Thus, the aim of this study was to evaluate the effects of filler content on mechanical properties of dual-cured resin cements light-cured immediately or after five minutes. The hypothesis was that the viscosity alteration promoted by alterations on filler content affects the possible effects of the moment of light-activation on the mechanical properties.

Material and Methods

Study design

This investigation was conducted using a 3 × 2 factorial study design to evaluate the 'filler content' on three levels (60, 65 or 68%) and 'moment of light-curing' on two levels (immediately or 5 min after cement mixing). The response variables evaluated were the flexural strength (σ_f) and flexural modulus (E_f) and Vicker's hardness (VHN).

Formulation of the experimental dual-cured resin cements

A model dual-cured resin luting agent was formulated using the monomers 2,2 – bis[4–(2-hydroxy-3-methacryloxyprop-1-oxy)pheny1]propane (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) at a 1:3 mass ratio. The monomers were obtained from Esstech Inc. (Essington, PA, USA). Silanated barium borosilicate glass fillers, 2 µm in average diameter (Esstech Inc.), were added at 60, 65 or 68% (*w/w*). The cement consisted of two pastes, one labelled base paste and another labelled catalyst paste. Camphorquinone (0.8 mass%) and diethanol-*p*-toluidine (3 mass%), both from Esstech Inc., were added to the base paste as the photoinitiator and co-initiator. Benzoyl peroxide (Vetec, Rio de Janeiro, RJ, Brazil) was added to the catalyst paste at a 3 mass% as a self-activated initiation system. Butylated hydroxytoluene (0.2 mass%) was added to both pastes as a radical scavenger.

Viscosity measurement

The viscosity measurements were performed using a digital viscometer LVDV-II+Pro (Brookfield, Middleboro, MA, USA) used in oscillatory mode with the following settings:

temperature of 20°C, speed of 5 rpm, shear rate of 10 Hz and run time of 60 s. Three measurements were conducted for each experimental condition.

Specimen preparation for the 3-point bending test

Specimens were prepared according to the bar-shaped dimensions specified by the ISO 4049 standard (19). Equal volumes of base and catalyst pastes were mixed for 15 s and inserted into a metallic split mold 25 mm in length, 2 mm in width and 2 mm in height. The material was covered by an acetate strip and light-activation was performed using three identical light-emitting-diode (LED) light-curing units (Radii-Cal; SDI, Bayswater, Victoria, Australia) with 1200 mW/cm^2 irradiance each activated. The tips of light-curing units were positioned 1 cm away from the specimen, distributed at equidistant points from the center of the bar. The light-activation was performed with three simultaneous light exposures for 60s. Following, the specimens were removed and the opposite side was light-cured in the same way. The cured specimens were wet-polished with #1200-grit SiC papers and stored in distilled water at 37±1°C for 24 h in the dark.

Three-point bending test

The dimensions of the bars were checked with a digital caliper accurate to 0.01 mm (Mitutoyo Corporation, Tokyo, Japan). The specimens were positioned in a 3-point bending device coupled to a mechanical testing system (Instron 3367, Instron Corp., Canton, MA, USA). The distance between supports was 20 mm and the load was applied to the center of the specimen. The diameter of both supports and of the loading rod was 2 mm. The tests were performed at a crosshead speed of 0.5 mm/min until failure and were monitored by the testing machine software (Bluehill 2, Instron Corp.). To calculate σ_f (MPa), the following equation was used:

$$\sigma_f = \frac{3Fl}{2bh^2} \text{ (Eq. 1)}$$

where F is the maximum load (N) exerted on the specimen, l is the distance (mm) between the supports, and b is the width (mm) and h the height (mm) at the center of the specimen. The E_f was calculated using the following equation:

$$E = \frac{F_1 l^3}{4bdh^3} \text{ (Eq. 2)}$$

where F_1 is the load (N) exerted on the specimen and d is the deflection corresponding to the load F_1.

Vickers hardness

Five fractured specimens from three-point bending test were randomly selected for hardness measurement. The specimens were included in epoxy resin cylinders and wet-polished with 180, 320, 400, 600 and 1200 grit SiC abrasive papers. Hardness measurements were performed with a Vickers indenter (HMV-2, Shimadzu, Tokyo, Japan) used under 1 KgF of load applied for 15 s. The indentations were measured and the values automatically converted to Vicker's hardness number (VHN). Six indentations were performed by specimen and average used for the statistical analysis.

Statistical analysis

Data for the σ_f, E_f and hardness were individually submitted to 2-way analysis of variance (ANOVA). All pairwise, multiple comparison procedures were performed using the Tukey's method ($\alpha = 0.05$).

Results

All results are displayed in Table 1.

Viscosity

ANOVA showed that the filler content significantly affects the resin cement viscosity ($P < 0.001$). The resin cement with 68% of filler content showed the highest viscosity and those with 60% the lowest values of viscosity.

Flexural strength

ANOVA showed significant effect only for the factor 'filler content' ($P < 0.001$), whereas the factor 'moment of light-curing' ($P = 0.215$) and the interaction between the factors ($P = 0.057$) were not significant. Resin cements with 65% of filler showed the highest σ_f, while the adding of 68% of filler to resin cement resulted in the lowest values of σ_f.

Elastic Modulus

ANOVA showed significant effect only for the factor 'filler content' ($P = 0.030$), whereas the factor 'moment of light-curing' ($P = 0.247$) and the interaction between the factors ($P = 0.908$) were not significant. The addition of 65% filler to cement resulted in a higher E_f than when adding 60% filler. Resin cements with 68% of filler presented intermediate values of E_f and without significant difference from the filler contents.

Vickers Hardness

Only the factor 'filler content' significantly affected the values of hardness ($P < 0.001$). The factor 'moment of light-activation' ($P = 0.308$) and the interaction between factors ($P = 0.477$) were not significant. Irrespective the moment of light-activation, 68% of filler resulted in the highest values of hardness. No difference was observed between the other filler contents.

Discussion

The present study hypothesized that a possible alteration on filler content followed by modification of cement viscosity could affect the effects promoted by moment of light-curing on mechanical properties. This was expected due to the viscosity of material affecting the mobility of reactional media. Thus, the speed of polymerization reaction chemically-activated can be reduced by increased viscosity, whereas the effects of delayed light-activation on polymerization process could be altered (20, 21). According to this idea, increasing the filler content of experimental resin cements was demonstrated to significantly increase their viscosity. The alteration of filler contents from 60 to 65% increased 6-fold the viscosity, while the cements with 68% of filler presented 10-fold more viscosity than those with 60%. However, despite these significant differences in viscosity between the experimental cements evaluated, no effects of moment of light-activation on mechanical properties were observed. Thus, the hypothesis of the study was rejected.

Irrespective of the moment of light-activation, the filler content significantly affected the mechanical properties. It was expected that increasing the filler content would result in improvements on all mechanical properties; however, this effect was not observed for σ_f and E_f. Increasing the filler content from 60 to 65% was followed by an increase on σ_f according to expected results. Conversely, adding more filler to obtain a cement with 68% of filler resulted in significant decreasing of the σ_f. Another previous study (Kim et al. 2002) [22] found similar behavior increasing the filler content of resin cements. The addition of filler commonly increases the mechanical properties of resin-based material until a limit of filler content (22). Following the addition of filler content can make the material prone to fracture because fillers may act as areas of stress concentration in this condition, leading to failure propagation and reduced fracture strength (22). However, it is important to emphasize that the values of the σf (approximately 67 MPa) reached by cements with 68% of filler (lowest values) was superior to the minimum values required by ISO (19) standardization (50 MPa).

Another unexpected outcome was observed for E_f measurements. Cements with 65% of filler content presented higher E_f than those with 60%, as expected. However, increasing to 68% the filler content did not result in improvements on E_f. Conversely, the values of E_f

observed for cements with 68% of filler were similar to those presenting 60% of filler content. The development of E_f has an important relation with the polymerization process, whereas slower polymerization reaction can be commonly associated to a reduction in the E_f (12, 15,16). The addition of 68% of filler is followed by significant increase of cement viscosity, reducing the mobility of reactional media. This slower polymerization reaction results in polymer with reduced density of cross-links and lower E_f (23,24). This effect of polymerization reaction speed on E_f could be expected also for delayed light-activation (14). However, the effects of the postponement of the light-curing on polymer formed can be less pronounced.

Regarding the hardness, a tendency was observed toward increasing the hardness of cements within the addition of filler, while 68% of filler content resulted in highest values. The hardness is the abillity of the material to resist the penetration of another material when the harder material will penetrates the surface of the other. The addition of filler, which is harder than organic matrix, results in ever increasing hardness of resin-based material (25). Similarly to other mechanical properties evaluated, the effect of filler content on hardness was not dependent from the moment of light-curing. It has been demonstrated that the moment of light-curing can affect the mechanical properties of cements and this effect depends on material composition. Based on the findings of the present study, the filler content seems does not affect this material-dependent behavior. Thus, further studies evaluating other components of dual-cured resin cements are necessary to determine the factors that can intervene in association between moment of light-curing and mechanical properties.

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Tables

Table 1. Results in means (95% Confidence interval).

Filler content	Viscosity (Pas)	Flexural strength (MPa)			Elastic Modulus (GPa)			Vickers hardness (VHN)		
		Moment of light-curing		Pooled average	Moment of light-curing		Pooled average	Moment of light-curing		Pooled average
		Immediate	Delayed		Immediate	Delayed		Immediate	Delayed	
60%	1.0 (1.0 - 1.1)	94.9 (79.1 – 110.7)	91.0 (79.3 – 102.7)	92.9^{B} (79.5 – 106.3)	6.8 (6.3 – 7.2)	7.1 (6.3 – 7.9)	6.9^{B} (6.2 – 7.5)	40.8 (37.9 – 43.6)	43.4 (39.1 – 47.7)	42.1^{B} (40.0 – 44.2)
65%	6.1 (5.9-6.4)	108.4 (84.5 – 132.3)	116.3 (101.8 – 130.7)	112.3^{A} (93.0 – 131.6)	7.6 (6.9 – 8.3)	8.4 (7.3 – 9.5)	8.0^{A} (7.0 – 9.0)	44.1 (42.0 – 46.1)	45.8 (43.9 – 47.7)	44.9^{B} (43.8 – 46.1)
68%	10.0 (9.1-11.0)	79.2 (71.6 – 86.8)	55.0 (37.6 – 72.4)	67.1^{C} (49.8 – 84.4)	7.2 (5.4 – 9.0)	7.7 (6.4 – 9.0)	7.4^{AB} (6.0 – 8.9)	49.6 (46.8 – 52.4)	48.9 (46.6 – 51.1)	49.2^{A} (47.8 – 50.6)

For pooled average, distinct letters indicate statistical differences ($P < 0.05$).

5. CONSIDERAÇÕES FINAIS

O conteúdo de carga afetou as propriedades mecânicas dos cimentos resinosos experimentais duais, independentemente do momento da fotoativação. O uso de 65% (em massa) de partículas de carga resultou nos maiores valores de resistência flexural e módulo de elasticidade. Já o cimento com 68% de carga teve os maiores valores de dureza.

6. COMUNICADO DE IMPRENSA

Algumas restaurações dentárias são confeccionadas extra oralmente em laboratório e, posteriormente, cimentadas ao dente preparado pelo cirurgião-dentista por meio de cimentos a base de resina. Estes cimentos endurecem (polimerizam) por meio de uma reação em que unidades menores (monômeros) se agrupam para formarem polímeros, sendo que a reação resulta em redução volumétrica (contração) do cimento. Esta contração volumétrica pode resultar em fendas nas margens da restauração, e em trincas no dente e na restauração, com redução de longevidade do procedimento restaurador. Um ponto importante é que a ativação da reação de polimerização destes cimentos se dá de duas formas: a partir da mistura dos seus componentes (ativação química) e da ativação por luz (fotoativação). A fotoativação destes cimentos é essencial para aumentar a sua resistência mecânica. Além disto, tem sido demonstrado que o aumento do tempo de espera entre a sua realização e manipulação do cimento pode reduzir os efeitos negativos da contração do cimento. Entretanto, dependendo da marca comercial do cimento, este retardo na fotoativação pode reduzir a resistência mecânica do cimento.

A dissertação apresentada pela discente Ana Carla de Assunção Oliveira, orientada pelo Prof. Dr. André Luis Faria e Silva, avaliou o efeito da concentração de cargas inorgânicas, adicionadas aos cimentos para aumentar a resistência, sobre os efeitos promovidos pelo momento da fotoativação em propriedades mecânicas de cimentos. Os resultados mostram que a concentração de carga afetou as propriedades dos cimentos, sendo este efeito independente do momento em que se realizou a fotoativação do cimento. Avaliou-se a resistência à flexão, quando amostras dos cimentos são flexionados até a fratura, rigidez destes cimentos (denominado de módulo de elasticidade) e a dureza, que é a capacidade de resistir ao desgaste por outros materiais. Para a resistência à flexão e rigidez, o aumento da carga elevou os valores destas propriedades até certo ponto, a partir do qual a adição de maior quantidade de carga reduziu a resistência e rigidez. Já para dureza, o aumento da carga resultou em maior resistência ao desgaste. Este estudo fez parte de um projeto maior do grupo de pesquisa coordenada pelo Prof. André, que também avaliou outros componentes dos cimentos à base de resina sobre os efeitos da fotoativação tardia.

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